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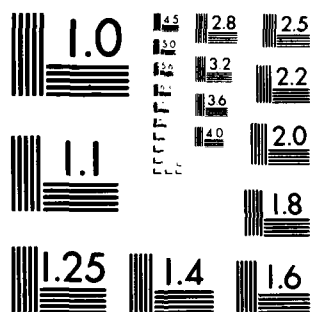
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The overall objective of this two-phase program was to develop techniques for fabricating high resolution liquid crystal reticles suitable for military application. The first phase of this program consisted of three technology development tasks, namely substrate improvements, liquid crystal seals, and		

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electronics seals. The goal of each development task was successfully accomplished. Results are summarized below:

1. Techniques were developed to produce 98 percent working electrode lines without repair (100 percent with repair) on LCR plates with a density of 580 lines per inch. Results were verified by optical inspection and electrical test.
2. LC cells fabricated with Ablefilm 539 sealant have now surpassed 7000 hours at +80°C and -17°C without cell deterioration. Additional cells with bulk electrodes and continuous AC voltage have surpassed 6200 hours at +25°C without alignment change. Other cells fabricated with Ablefilm 539 showed no signs of weakening when exposed to liquid nitrogen or boiling water conditions.
3. A dual coating of silicon nitride and parylene demonstrated moisture protection, but the process was costly to apply and difficult to repair. Conventional hybrid solder sealing technology was used to devise two alternate packaging concepts to maximize producibility and maintainability with potentially lower production costs.

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PREFACE AND ACKNOWLEDGEMENTS

Technology development of techniques for fabricating high resolution liquid crystal reticles was conducted by the Hughes Aircraft Company, Culver City, California, from October 1978 through September 1979, under contract DAAK-10-78-C-0386 for the U.S. Army Armament Research and Development Command, Dover, New Jersey. This interim technical report relates to that effort.

Mr. Robert Shipe served as the Contracting Officer's Representative for the program. Program Manager for Hughes Electro-Optical Engineering Division was Mr. Barry C. Gilbert. Particularly valuable contributions were made by Hughes Research Laboratories (HRL) and Technology Support Division (TSD). Dr. J.D. Margerum and Dr. G.D. Myer, from HRL, provided liquid crystal seal experience and consulting services on liquid crystal materials. Dr. H. Garvin provided consulting services on substrate improvements. Dr. P.Y. Hsieh and Mr. T. Ammons, from TSD, provided support for substrate improvements and liquid crystal seals. Messrs. G. Dryer, L. Roveda, and K. Yamamoto supported the electronic seals effort.

SECTION I

INTRODUCTION AND SUMMARY

The liquid crystal reticle (LCR) is an electronically-operated digital moving reticle mounted directly into the optical train of a sight. The LCR has many important advantages over state-of-the-art servo-driven opto-mechanical projected reticle, including:

1. Digital design and simple computer interface
2. Much smaller size and weight
3. Stable boresight and excellent linearity
4. No moving parts and much better reliability
5. Lower acquisition and maintenance costs, both for the device itself and for the system of which it is a part
6. Improved light transmission as beamsplitters are eliminated.

In all, the LCR is a much more attractive device.

The feasibility of the LCR has been demonstrated with hardware models delivered both to the Army and internally to Hughes. The Hughes LCR was integrated into a modular fire control system in January 1977 and was widely demonstrated. This reticle, approximately 4.5 x 4.5 x 0.5 inches in size, fits directly in the optical train of the M32 8X day elbow. The deflection capability as a disturbed reticle is +69 to -17 mils in elevation and ± 30 mils in azimuth. The center-to-center spacing of 0.2 artillery mil between lines gives a reticle accuracy of 0.1 mil (1σ), which is slightly better than the current M60A3 moving reticle system. Incorporated into the LCR, in the telescope field-of-view, is an alphanumeric display of laser range, ammunition type, and other discrete symbols. The assembly is currently operational having 95 percent total working electrode lines in the azimuth and elevation axes (approximately 98 percent working lines per axis).

Results from a recent Army contract proved that an LCR can be made to withstand the armored vehicle environment. The results of that effort are summarized below.

1. Delivery of a prototype LCR with 49 lines for elevation and 299 lines for azimuth
2. A transparent thin film heater that allows operation from -40 to +125°F
3. Shock and vibration mounting scheme for armored vehicle environments
4. A new Hughes developed electronic driver scheme with simple electronic controller
5. Installation and operation on a thermal nightsight, using essentially the same volume as the existing fixed reticle, and with only one reticle line not functional.

The overall objective of this two-phase program was to complete the technological evolution of the LCR by developing techniques to fabricate high resolution LCRs suitable for the tank environment. Phase I consisted of three technology development tasks, while Phase II will consist of hardware delivery. The goals for each development task were to:

1. Develop techniques to achieve 100 percent working electrode lines on LCR plates with a density of 580 lines per inch.
2. Develop a seal for liquid crystal (LC) cells that is chemically compatible with LCR manufacturing.
3. Develop a seal for the electronics required to address and drive individual reticle lines compatible with LCR manufacturing.

The three development tasks were successfully concluded. The results are summarized below:

1. Techniques were developed to produce 98 percent working electrode lines without repair (100 percent with repair) on LCR plates with a density of 580 lines per inch. Results were verified by optical inspection and electrical test.
2. LC cells fabricated with Ablefilm 539 sealant have now surpassed 7000 hours at +80°C and -17°C without cell deterioration. Additional cells with bulk electrodes and continuous AC voltage have surpassed 6200 hours at +25°C without alignment change. Other cells fabricated with Ablefilm 539 showed no signs of weakening when exposed to liquid nitrogen or boiling water conditions.
3. A dual coating of silicon nitride and parylene demonstrated moisture protection, but the process was costly to apply and difficult to repair. Conventional hybrid solder sealing technology was used to devise two alternate packaging concepts to maximize producibility and maintainability with potentially lower production costs.

This interim technical report documents the techniques developed for fabricating high resolution liquid crystal reticles. It is organized into three sections: Section II - Substrate Development Task, Section III - Liquid Crystal Seal Development Task, and Section IV - Electronic Seal Task.

SECTION II

SUBSTRATE DEVELOPMENT TASK

2.1 AIMS AND OBJECTIVES

The overall objective of this task was to achieve 100 percent working electrode lines on liquid crystal reticle glass substrate plates. Fabrication of defect-free electrode lines, with an approximate density of 580 lines per inch, required that a technique be developed from investigations of the following processing procedures:

1. Photomasks
2. Substrate Preparation
3. Metallization
4. Photoresist
5. Etching.

2.2 RESULTS

2.2.1 General Procedures

Clean room conditions were used for substrate preparation, metallization, and photolithographic pattern generation. To reduce surface aberrations to the absolute minimum, each procedural step of the overall process was divided into two or three procedural substeps. Visual inspection and thorough physical cleaning were implemented between each substep. The clean room used for substrate improvements is shown in Figure 1.

2.2.2 Photomasks

Past LCR patterns were generated by a reliable photo-lithography technique used in the semiconductor industry since the 1950s. Basically, an accurately dimensioned set of drawings is generated by a mechanical designer for each azimuth and elevation plate. Three or more layouts using different scale factors are required for each reticle plate (e.g., liquid crystal cell, chip bonding pad, chip and signal electrical interconnect). These layouts require precise design since each must fit into a previously established area.



Figure 1. Clean room used for substrate improvements.

The layouts are digitized on a Calma interactive graphics system to generate composite plots. Each composite plot is checked for errors, and corrections are incorporated immediately. Then a 10X master is generated on a Gerber photoplotter and checked for correct exposure and open or shorted lines. The 10X photophots are utilized to generate 1X chrome on glass photomasks which then are utilized to contact print the pattern onto a metallized glass reticle substrate.

Specialized vendors are used to obtain the 1X chrome photomasks. Several vendors have the potential to produce the high quality photomasks required on this program. Two vendors, Optronics and Micromask, were investigated further; Optronics was selected to produce the chrome photomasks.

Two (positive and negative) working sets of defect-free* chrome masks were obtained from Optronics to obtain high line yield; one mask is shown in Figure 2. The masks were made from Hughes 10X masters and were anti-reflection coated, a technique successfully used in semiconductor wafer fabrication, to enhance contrast or reduce ghost images. Optronics was selected for continued needs on this program. The quality of photomasks obtained from Optronics is shown in Figure 3.

Also Micromask could supply high quality chrome masks by photographic camera reduction and contact printing with Hughes 10X masters. Micromask also suggested two other approaches for making defect-free chrome masks (pattern generation and E-beam). They could pattern generate a chrome master from a Hughes Calma LCR library tape via converting to David Mann format with mask defects corrected by a Florod Mask saver. No working masks were procured from this source, because Optronics proved satisfactory.

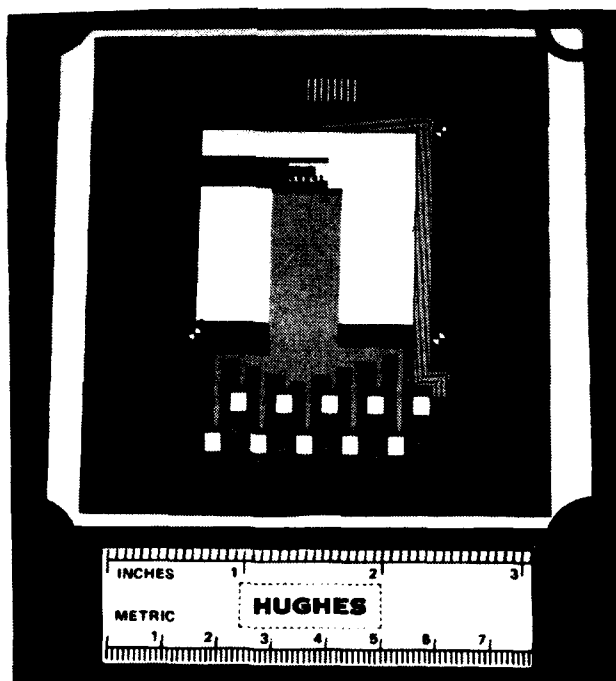
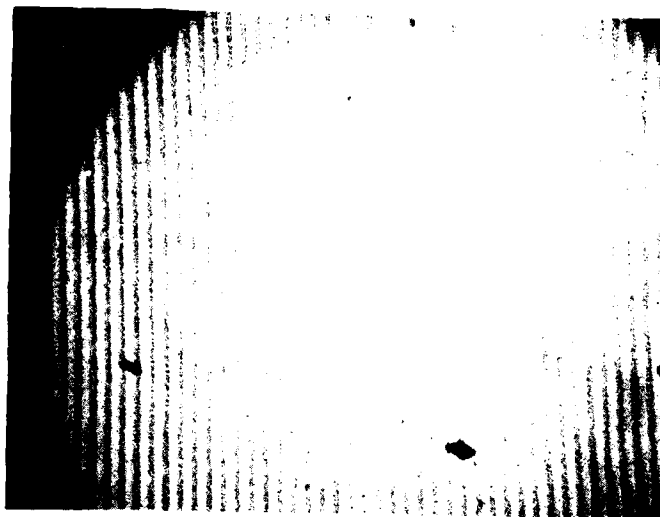


Figure 2. High quality chrome photomask used to obtain 96 and 98 percent yield substrates.

*Defect-free means no open lines, no line-to-line shorts, or pinholes larger than one-third a line width.



a. 40X — even lines, no scratches.



b. 160X — high quality lines.

Figure 3. Magnification of high quality chrome photomasks produced from Hughes 10X masters.

2.2.3 Substrate Preparation

Transparent conductive glass (indium tin oxide (ITO) coated soda lime glass) was purchased from Optical Coating Laboratory, Inc. (OCLI), Santa Rosa, CA. The manufacturer's designation of the coating was LCM 4000. The size of the glass was 11 x 16 inches and the coating had a conductivity of 10 to 30 ohms per square. Both 1/4 and 1/8 inch thick substrates were procured.

The stock sheet was scribed and broken into 2-1/2 x 3-1/2 inch substrates. Caution was used to ensure that the conductive coating was not scratched by the flaked-off glass chips. Then the cut sheets were waxed together into a block and ground to the desired dimensions. The figured substrates were then inspected under a microscope for pinholes and scratches in the reticle area.* Acceptable substrates were vapor degreased in TCE, isopropanol, and then sent for metallization.

2.2.4 Metallization

Outside the LC cell area, metallization over the ITO layer is necessary to enable bonding the electronic driver chips and to provide electrical interconnects. Three metal films (Ni-Cr-Au, Cr-Au, and Mo-Au) and three metallization machines (E-beam, RF sputter, and hot filament) were investigated. The best results were obtained with Ni-Cr-Au film using E-beam deposition.

Each metal film used approximately 150 Å of Ni-Cr or Cr with 5000 Å of Au; thickness was based on previous experience obtained in fabricating LCRs. Metallization was checked by testing adhesion with Scotch tape and pull strength of 1 mil Al wire bonds.

Using an Airco E-beam machine, 150 Å of Ni-Cr with 5000 Å of Au were deposited over the ITO layer, while keeping the substrate in circular motion for a uniform coating.

Initially, spikes in the film, which affect fine line resolution in the lithographic process, were observed in the E-beam evaporated Au film. The spikes were created by a fast deposition rate of Au during the deposition

*Pinholes and scratches in other areas were not critical, because they were covered with gold.

process and source contamination. When the rate was decreased and the source kept cleaner, no spikes were observed. Coatings produced by this method were smooth and dense. Samples passed the Scotch tape test and gave 6gs pull with wire bonds. Overall, this metallization system gave the most consistent results.

The same Ni-Cr-Au film was also deposited over ITO coated samples with a CVC hot-filament evaporator. Samples did not consistently pass the Scotch tape test, because of contamination of low vapor pressure materials in the vacuum chamber. An effort to clean the evaporator was unsatisfactory, and investigations of hot-filament metallization stopped.

RF sputtering was also investigated. Using a single target machine (a machine capable of holding only one metal target), approximately 120 Å of Cr (Ni-Cr target not available) was deposited first. Then the chamber was opened, the Cr target replaced, and approximately 5000-7600 Å Au deposited. Coatings produced by this method were smooth and more dense than the E-beam coatings. Samples passed the Scotch tape pull test and gave 8g pull with wire bonds. Although results appeared better, RF sputtering was not selected as an alternate to E-beam deposition because of the inconvenience of changing targets and chance of contamination.

2.2.5 Photoresist

Several photoresists evaluated for high resolution and high line yield included: Shipley positive resists AZ1350J and AZ1350B, Waycoat IC negative resist from Hunt Chemicals, and Riston negative film from Dupont. The Shipley AZ1350B was selected as the best photoresist for use on this program.

Riston (Dupont) film was somewhat difficult to laminate on the thick glass substrates. High resolution was also difficult to obtain because of the film thickness. This photoresist was not suitable for the LCR.

Waycoat negative IC resist (Hunt Chemicals) was spin coated on test substrates at 2000 rpm. The main drawbacks of this resist were the organic solvents required for development and less exposure latitude than the Shipley positive resists. This photoresist was also not suitable for the LCR.

Shipley AZ1305J, a positive resist, was spin coated on test substrates at 2000 rpm. An excessive buildup of photo-resist occurred around the

substrate perimeter due to high viscosity of the resist. The buildup was not eliminated by increasing the spin speed to 4000 rpm. The excessively thicker part of the photoresist could be removed by double exposure; i. e., covering the central area with an opaque mask and re-exposing to the UV light. Double exposure experiments were marginally successful, and the use of Shipley AZ1305J was not considered a sound choice for the LCR.

AZ1305B, being lower in viscosity, did not have a build-up problem. Because of low viscosity, however, the coating was thin, even at a preferred slow spin speed of 2000 rpm. The thin layer gave excellent line resolution but could not withstand the sputter etching process (sputter etching is preferable over wet chemical etching, to be described later). The problem was alleviated by a combination of chemical etching and sputter etching. Good fine lines with sharp definition were obtained with this approach, and Shipley AZ1305B was selected as the best available photoresist for the LCR. A set of procedures were established to apply Shipley AZ1305B to glass substrates and are briefly described next.

Before spin-coating, the substrate surface was rinsed with isopropanol while it was spinning. With this procedure the surface was cleaned, and it became easier to spread the photoresist. Next the photoresist was filtered through a 1 micron filter immediately before applying it to the substrate. Then the coated substrate was soft baked at 80°C for 30 minutes before exposure using a chrome mask. The photoresist spinner and exposure system are shown in Figure 4. The chrome mask was aligned to the substrate to obtain exposure for the required delineation. The mask surface nearest the light source was flooded continually with dry nitrogen during exposure to prevent debris from settling on the mask and imposing defects onto the final pattern.

Exposed resist was developed with 50 percent AZ developer and 50 percent DI water for about 60 seconds with constant agitation in the direction of the fine lines, then rinsed for 60 seconds in DI water, and blown dry with hot nitrogen before being hand-baked in air at 100°C for 30 minutes. Finally, the developed pattern was inspected thoroughly for defects, before etching.

A side advantage of using a positive resist was that dust particles, lint, and particulate contamination in the photoresist showed up as line-to-line



Figure 4. Photoresist spinner and exposure system.

shorts. Whereas, with a negative resist these contaminants showed up as an open line. Furthermore, a line short was much easier to correct than an open line.

2.2.6 Etching

Three etching techniques were studied: wet chemical, dry RF sputter, and a combination of wet chemical with dry RF sputter etching. The combination of wet chemical and dry RF sputter etching gave the best results.

Wet chemical etching tended to undercut the lines. This result was surprising because the ratio of line width (1.2 mils) to metallization thickness (0.02 mil) was sufficiently large to withstand some undercutting. The undercutting occurred while etching the ITO layer (rapid etching) and caused the Au to lift. In cases where the Au did not lift, uneven lines were observed. Wet chemical etching did not prove satisfactory for use with the LCR.

Dry RF sputter etching was solely dependent on the photoresist used. Best results were obtained with a thick photoresist layer, such as that encountered with Shipley AZ1305J. A thick photoresist layer withstood prolonged etching without deterioration, but gave edge build-up during application. In addition, the photoresist had to be vacuum baked for 1 hour before insertion in the MRC RF sputter etcher. This step was very important because without this prebake, the resist tended to outgass rapidly in high vacuum and generated small bubbles that spread into delineated areas to cause shorts. Because of the limited success with dry RF sputter etching, this approach was selected as an alternate to the preferred combination of wet chemical with dry RF sputter etching.

A combination of wet chemical etching with dry RF sputter etching was preferred for the Shipley AZ1305B photoresist. This approach was selected because of the thin photoresist layer that would not withstand prolonged sputter etching. The process established for the combination of wet chemical and RF sputter etching is briefly described next.

Only substrates with defect-free exposed lines (or nearly so) were selected for etching. Selected substrates were prebaked to eliminate possible bubbles forming in the photoresist during the later etching steps.

First, the selected and prebaked substrates were exposed to a gold etchant (J.E. Halma) until the exposed metallization was removed. Next, substrates were rinsed thoroughly and blown dry with nitrogen gas. Then the bottom of each substrate was greased (Apiezon M) to facilitate heat transfer and attached to a pallet. Substrates were RF sputter etched in a 12-inch MRC machine at 400 watts for 10 minutes with a low pressure inert (Ar) atmosphere. This process was repeated until the delineated areas were free of metal and ITO. After etching, the grease was removed with TCE (trichloro-ethylene) and the remaining photoresist rinsed off with Acetone. A modified Au etching process was used on test samples to open the transparent LCR active area.

2.2.7 Test Results

Four LCR azimuth substrates were prepared by cutting and grinding 1/4-inch thick ITO coated glass from OCLI. The ITO layer was overcoated with

Ni-Cr and Au by careful E-beam evaporation. The reticle pattern was generated on the metallized layer by using AZ1350B photoresist and a defect-free chrome mask supplied by Optronics. After development and hard baking, the substrate metallization was wet chemical etched. The last layer of ITO was sputter-etched in the 12-inch MRC sputter system. The photoresist was stripped after etching, and finished substrates were optically inspected and manually probed for shorts and opens in conductor lines. Results from processing without rework are given below:

<u>Substrate Number</u>	<u>Number of Shorts</u>	<u>Lines Involved</u>	<u>Opens in LC Cell Area</u>	<u>Working Line Yield, percent</u>
1	9	21	0	93
2	3	6	0	98
3	5	11	0	96
4	17	33	0	89

The line yield was computed on the basis of 301 lines. Two open lines were observed near one IC chip mounting pad (outside LC cell area) on Substrate 2. The opens were caused by a scratch in the photoresist that could have been repaired before etching. Most shorts could have been opened by a Masksaver.

Figure 5 shows 96 and 98 percent working electrode line substrates. Magnification of the etched conductor lines on the 98 percent yield substrate are shown in Figure 6 and may be compared to the similar chrome lines of Figure 3.

2.3 SUBSTRATE REPAIR

Although not part of this contract, substrate repair was considered an important aspect of the substrate improvement task. For large substrate quantities, the probability of obtaining 100 percent yield appears low because of the possibilities of contamination in process materials and environment, plus human errors. At the same time, the probability of producing large quantities of 96 or 98 percent yield substrates appears high. Thus, it

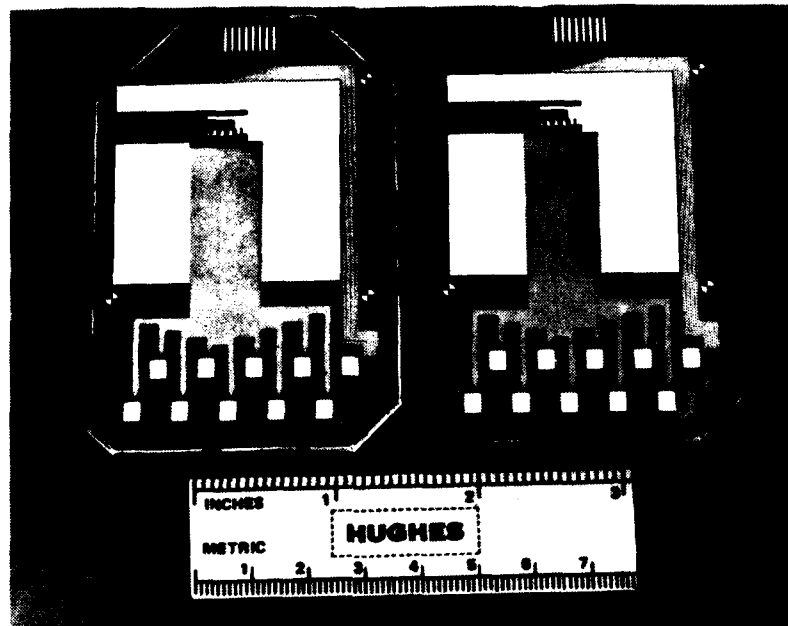
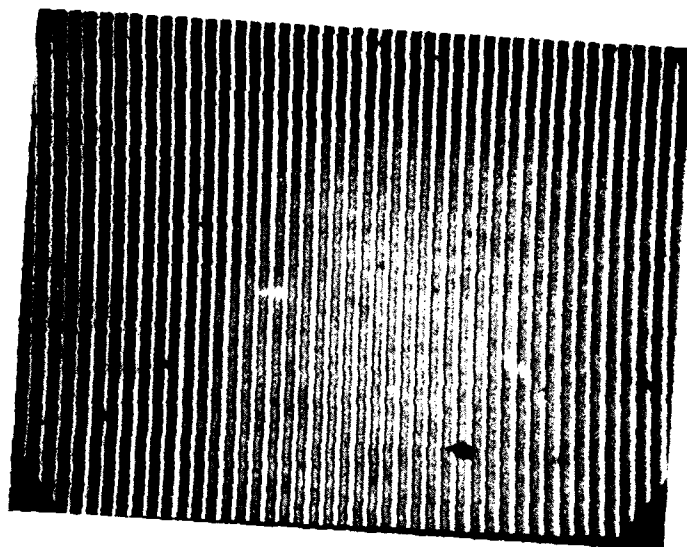


Figure 5. 96 and 98 percent working electrode line substrates.

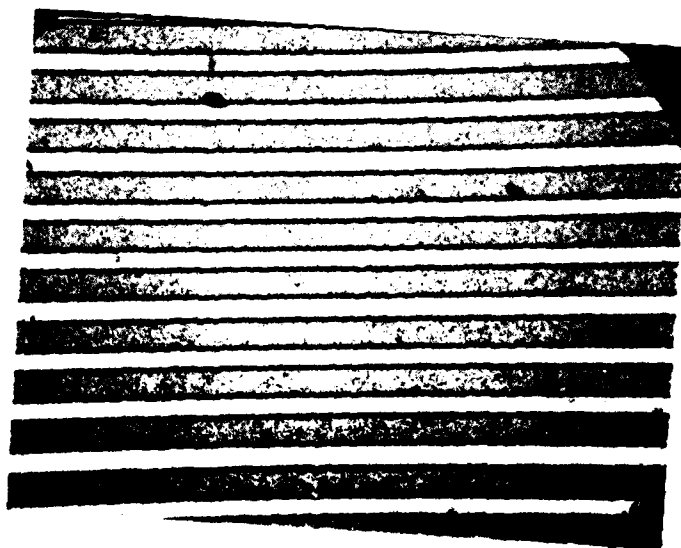
appears easier to correct the few defects on a large number of plates than to keep trying for 100 percent process yield. Capabilities for repairing few defective spots were investigated from a cost effective viewpoint.

Open lines in the active reticle area were not repairable because the conductors were ITO, and the scars from repair would cause defective liquid crystal molecular alignment. Fortunately, open lines were rarely encountered with the positive photoresist. Open conductors near the chip mounting areas were repaired by standard wire bonding; i.e., 1 mil Al wire was used to bridge an open in a 2 mil wide line.

Three methods were tested for repairing shorts, namely electric arc, YAG laser, and pulsed Xenon laser. The pulsed Xenon laser proved the best approach for substrate repair. An electric arc using the LCR substrate as one electrode to burn off the shorted spot was tested. The size of the burning area was not easy to control, and the burned scar was objectional. A diamond stylus was also useful in scribing open shorts, but scratches were left in the glass that were considered undesirable for shock loading. A Korad laser system (YAG laser, Mode KY3), designed for resistor trimming, was



a. 40X — etched substrate lines.



b. 160X — etched substrate lines.

Figure 6. Magnification of etched conductor lines on 98 percent yield substrate.

useful for opening shorts between lines with more than 0.001 inch spacing. The major drawback for this laser was that the beam size was not easily adjusted and the beam proved difficult to focus.

A "Mask saver" station, which used a xenon laser and was manufactured by Florod Corp., Hawthorne, CA., was more versatile and viable for the repair work. It could be fine focused, and the beam size adjusted freely to match the defect configurations. It also had a spot marker for the laser so that the operator could pinpoint the defective spot. Using this laser repair technique, substrates with process defects were finished with 100 percent line yield.

Using the "Mask saver" station, the time required to spot defects, zap them open, reclean the substrates, and retest for line yield was considerably less than the time required to process another batch of substrates for 100 percent yield. Therefore, the ability to salvage plates with few defects was judged highly cost effective.

2.4 CONCLUSIONS

The overall objective of this task was to achieve 100 percent working electrode lines with approximately 500 lines per inch density of liquid crystal reticle glass substrate plates. To achieve the objective of this task, techniques were developed from investigations of the process sets, namely photomasks, substrate preparation, metallization, photoresist, and etching.

A major effort to improve the quality of photomasks contributed considerably to the success of this task. The establishment of strict cleaning procedures for substrate preparation improved yield by reducing particle contamination. The use of careful E-beam metallization virtually eliminated voids and metal spikes, a contributor to poor line yield. Proper selection and application of photoresist was, possibly, the most important step in processing substrates. By properly exposing the photoresist and with visual inspection before etching, the line yield was improved. Finally, once the process was understood and the parameters defined, few defects were observed from etching. From the techniques developed on this task, plates of 96 and 98 percent working electrode lines were fabricated without repair.

Although not part of the contract, substrate repair was investigated from a cost effective view point. The time required to repair few defects (2 to 4 percent) on a substrate was judged considerably less than the time required to process a new substrate and attempt 100 percent process yield. Using pulsed Xenon laser techniques developed to repair defects, plates of 100 percent working line yield were fabricated.

SECTION III

LIQUID CRYSTAL SEAL DEVELOPMENT TASK

3.1 AIMS AND OBJECTIVES

The overall objective of this task was to define parameters and develop a seal that was chemically compatible with the reticle manufacturing process. To ascertain these parameters, tests were required on various peripheral sealant candidates, methods of filling the cell, sealing the fill hole, LC alignment, and bulk LC material – all to ensure long operational and storage lifetime. A sufficient quantity of material was required to allow parallel testing of heating, cooling and voltage effects on sealed cells. Finally life-tests were required on sample cells to prove sealant compatibility with the reticle manufacturing process and long lifetime.

3.2 RESULTS

3.2.1 Liquid Crystal Material

The first requirement was to synthesize more LC for testing. The Hughes proprietary LC-R9 material is a multi-component mixture, which is doped for conductivity. Each component was synthesized and recrystallized to obtain 99.8 percent purity, determined with an analytical liquid chromatograph. For a few components, however, recrystallization was not sufficient to achieve the high level of purity desired. For these samples, a preparative liquid chromatograph was used to separate the desired material for similar chemical structure impurities. The procedures for obtaining high purity LC components are described in Appendix A.

3.2.2 Liquid Crystal Sealant

To meet the objective of this task, the peripheral seal around the liquid crystal cell that comprises the active optical display must fulfill a variety of needs. It must contain the liquid crystal fluid over a wide temperature range and allow for the differential expansion coefficients between the glass substrates, the seal, and the fluid. It must be formed to allow filling the cell

after the seal is made. Means must be provided for plugging the filler hole(s). The seal must protect the liquid crystal material from moisture, oxygen, and other contaminants throughout the required life of the device. And, of course, the seal itself must not contaminate the liquid crystal. The desired life of the device is 10 years, of which several hundred to several thousand hours may be operating time.

Three types of peripheral sealants (thermoplastics, glass frits, and dry film preforms) were investigated for use in the LCR. The thermoplastics were unsatisfactory because of reaction with the LC material or poor screen print characteristics. The glass frit was not compatible with established LCR fabrication techniques because of the high (500°C) temperatures and extensive changes required for Hughes proprietary LC-R9 material. Excellent results were achieved with a dry film preform sealant, Abelfilm 539; it also satisfied the task objectives. Results from the three types of peripheral sealant are described in the remainder of this subsection.

Thermoplastic Sealants. The thermoplastic sealants were applied by a screen print process similar to that used in commercial art. Basically, the sealant was screen printed around both sides of each electrode plate with 0.3 to 0.5 mil thickness using 325 mesh screen and zero emulsion thickness. Then, the printed sealant was baked at +125°C for up to 1 hour to "cure" the polymer and drive out any solvents. Next, LC material (LC-R9) was dropped on one electrode plate and covered with the other electrode plate to form a captive cell. Finally, each cell was clamped together and heated (>100°C) for a few minutes. The advantages of these procedures were simplicity of sealing and no fill hole.

A thermoplastic sealant TPA-82 (American Liquid Crystal Chemical Co.) was evaluated first because it appeared to have the most potential and was widely used in industry. TPA 82 was easily screen printed on test cells and required a 120°C temperature for sealing. Test cells functioned well electrically. However, storage at 80°C for about 1 day showed creeping of the sealant into the viewing area and misorientation of molecular alignments along the perimeter because of interaction between sealant and LC. On further investigation, TPA 82 was found to have a softening point below 120°C

and to contain plasticizer. Destruction of the alignment was attributed to the plasticizer.

Other screen print thermoplastics investigated were Eponol 53-L-32 (Shell Oil), Methylon 75202 (General Electric), 642-1 and 681-14 (Ablestik Laboratories). All sealants were difficult to screen print, and the solvent odor was highly objectionable. Two thermoplastics, AL 600 and MC 600 (Mermid), obtained from the Hughes Adhesive Laboratory were also tested but showed poor screen printing characteristics, and the seal was much too brittle. No thermoplastic sealant was judged suitable for use in the LCR, and all work on these sealants was stopped.

Glass Frit Sealants. The glass frit sealing process investigated was quite similar to the thermoplastics sealing process. A paste of glass beads was screened onto one of the glass sheets in the form of a ring seal. After glazing, the second plate was put into place, and the assembly heated to fuse the glass into a solid seal. The major differences were that the frit processing temperature was significantly higher (500°C), and a fill hole was required. Even though a glass frit seal with a soldered fill hole was considered a hermetic seal, the high temperature process impacted fabrication of an LCR to a greater degree.

As reported earlier, the Ni-Cr-Au or Cr-Au metallization was unsuitable for high temperature sealing because of interdiffusion between metal layers. Although encouraging results were obtained from some initial work on a Mo-Au metallization system, a change (factor of two) in the resistivity of the ITO occurred during heating and had to be controlled by limiting the time at high temperature. In addition, the Hughes proprietary LC-R9 material was impacted by a glass frit seal because the dopant and surface alignment requirements were not compatible.

A sizeable expanded effort was needed to further investigate the compatibility of using a glass frit sealant in the present LCR manufacturing process required and was considered beyond the scope of this contract. At this same time, successful sealing was achieved with a dry film preform, and work on the glass frit seal was stopped.

Dry Film Sealant. The dry film peripheral seal is formed from a thin piece of material (usually Mylar), which is coated on both sides with a film of epoxy. The total thickness is accurately controlled, and the epoxy is specially formulated for use with LC materials. The composite sheet is cut to shape and clamped in place between the two pieces of glass as shown in Figure 7. Then the whole assembly is brought to a curing temperature (approximately 125°C), and the seal is formed.

Ablefilm 539 (Ablestick Laboratories) was the only dry film peripheral sealant investigated. Its test was strongly recommended by Hughes personnel who had experience with this material as well as experience with LC devices.

Appendix B contains a description and specification on Ablefilm 539. Hughes successfully applied Ablefilm 539 to early watch LCDs, while the first use in an LCR occurred on a previous Army contract (DAAK-70-78-C-0023). During that contract, an Ablefilm sealed LCR was subjected to shock and vibration tests with no damage. A sealed cell was also temperature cycled from -40 to +51°C with no leaks.

Extensive testing was used to determine the applicability of Ablefilm 539 for LCRs. Initially several cells were constructed of both type I and type II material, then filled with Hughes proprietary LC-R9. Some cells were stored at +80°C, while others were stored at -15°C. The type I cells deteriorated after 200 hours at +80°C, while the type II cells did not deteriorate. Additional cells were constructed of both types of material (type I and type II), then filled with Hughes proprietary LC-R9, and the tests rerun. Again the type I material failed in +80°C storage. Little evidence was uncovered to indicate the cause of the type I cells failure in +80°C storage.

Results from the life tests (subsection 3.2.4) have been very encouraging because sealed cells using Ablefilm 539, Type II, material have now surpassed several thousand hours without deterioration. Two additional tests were conducted on cells fabricated with Ablefilm seals. One cell was cooled with liquid nitrogen in an attempt to break the Ablefilm seal. The glass cracked, and the seal remained intact. A second cell was soaked for 1 day in boiling water and 6 additional days in room temperature water. At no time did the sealant show signs of weakening.

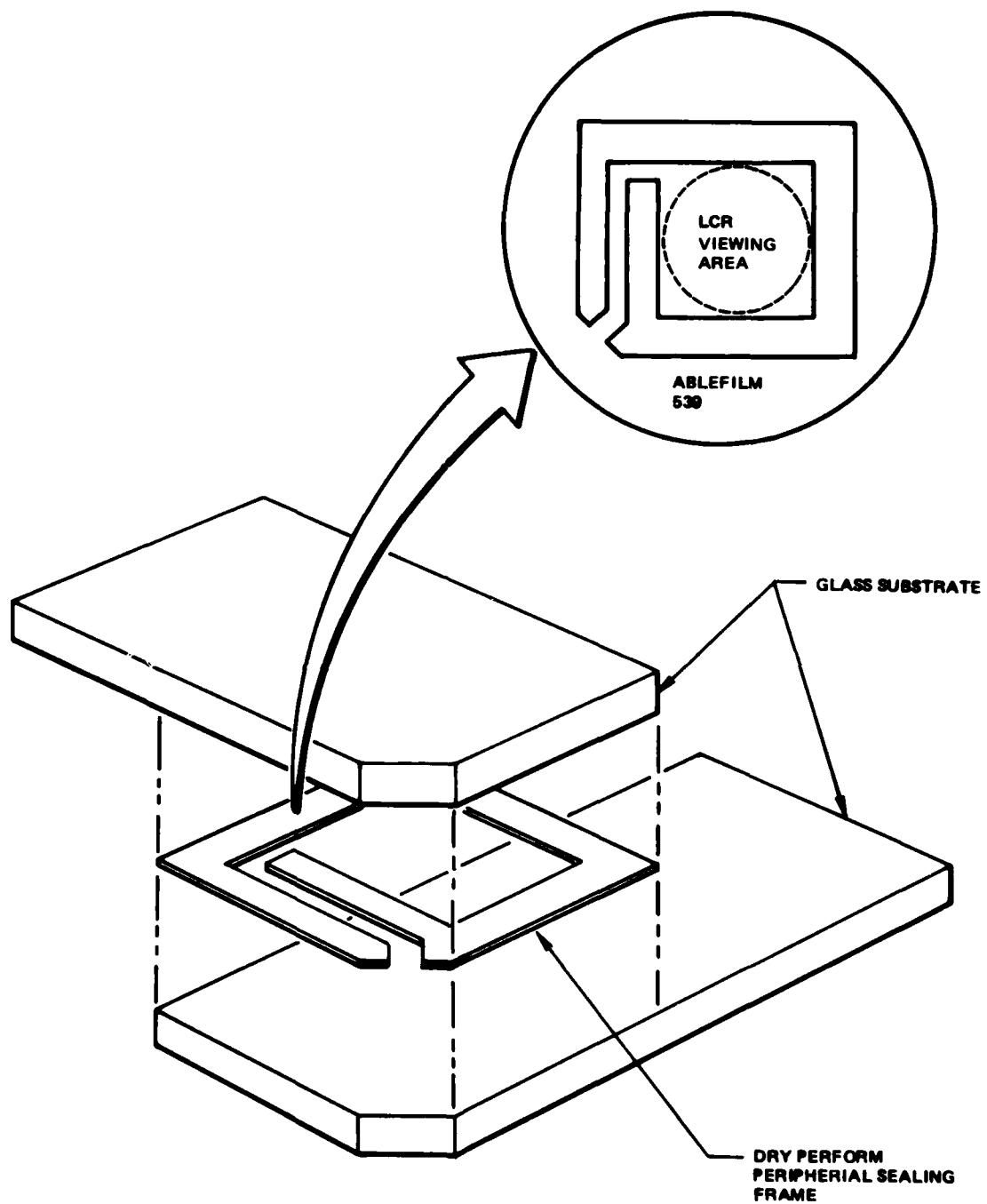


Figure 7. Liquid crystal cell assembly using dry film peripheral sealant with fill channel.

3.2.3 Fill Hole and Fill Hole Sealant

The method of filling the LCR cell and plugging the fill hole(s) were considered important subtasks to prevent leaks. Two techniques were investigated to fill an LCR. The one-hole edge fill method was preferred over a two-hole method.

Two fill holes were investigated for use on the LCR because some commercial devices still use the technique. The technique does not require special fill apparatus (vacuum system) because the LC material is forced through the first hole, while air exhausts through the second hole. Problems with forcing the LC material into the cell, plus a much higher probability of leakage (two holes) forced the abandonment of this technique.

Early commercial LC displays, including the first LCRs, used a hole drilled through glass for filling in a vacuum chamber. Commercial LC devices, including the LCR, later went to a more economical edge fill method that utilized the LC peripheral sealant (see Figure 7) and a vacuum chamber. Briefly, the LCR assembly is placed in a vacuum chamber and, after evacuation, dipped into a pool of LC material. At first capillary action starts filling the cell, but as the vacuum is slowly released, LC material is sucked into the remaining evacuated cell area. Alternately, to conserve LC material, the fill hole can be oriented so that a drop of LC material covers the opening, then as the vacuum is slowly released the drop is sucked into the evacuated cell.

Three approaches were investigated to plug the fill hole(s): indium, epoxy, and solder. An epoxy plug performed satisfactorily and was preferred over the other two approaches.

Indium is soft, easily forced into a small glass hole, and does not react with most LC materials. Early LCRs used an indium plug for a drilled glass fill hole. However, greater contraction of the indium at -40°C , during temperature cycling, created leakage and required epoxy reinforcement. Thus the value of an indium plug at low temperatures was useless; therefore, this approach was judged not suitable for use on the LCR.

A solder seal technique studied, relied on Ni-Cr-Au being evaporated around the fill hole and solder applied to seal the hole. Although this seal was more expensive than epoxies, it was also considered a hermetic

seal. One major drawback was the necessity to vacuum deposit Ni-Cr-Au around the LCR fill hole. The solder seal technique to seal a fill hole was not tested on LCR sample cells because of the success achieved with the use of epoxies.

Epoxy fill hole sealants were used on some past LCR and on all test cells constructed for this contract. Two Ablestik sealants, 224-1 and 408-3, performed satisfactorily as an edge-sealant. Both were single component room temperature curing epoxies. The difference between the two was in the working life, 20 minutes for the former and 2 hours for the latter. The sealed cells showed no leakage and interaction with the LC after temperature cyclings and storage at 80°C for an extended length of time.

3.2.4 Life Tests

Test cells used for life tests were assembled by the procedures outlined in Appendix C. The majority of AC type cells were prepared with bulk electrodes, i.e., continuous electrodes covering the LC cell area. Three major life tests were selected for sample cells: +80°C storage, -17°C storage, and continuous AC voltage drive (100 Hz). Hot storage at +80°C was selected for an extra margin of safety over the +71°C maximum storage temperature specified in a tank environment. The -17°C temperature was selected as the point where bulk crystallization of Hughes proprietary LC-R9 probably would occur. This temperature was more critical than storage at -40°C (or lower). Finally, a continuous AC voltage drive test was selected to study long term alignment conditions in the LC cell.

The heating lifetest was monitored by observing alignment changes in the sealed cells after storage in an oven set at 80°C. Each cell was observed between crossed polarizers, which proved to be a very sensitive test. After more than 6000 hours, the LC alignment had no observable changes.

The freezing life test was monitored the same way as the heating test with storage at -17°C. Again, after more than 6000 hours, no change in alignment or LC crystallization was observed in cells between crossed polarizers.

Two methods were used to test the effect of voltage on the LC alignment test cells. In one case, continuous bulk electrodes were used in sealed cells. In the second case, a small test reticle was made with fixed lines addressed by a continuous voltage. In each case, a 20-volt (100 Hz) squarewave signal was generated with the same type of chip used in the reticle. For the continuous bulk electrode cells, no change in alignment was observed after more than 6000 hours. A residual line was observed (without polarizers) in the reticle-type cell, however, after 1550 hours with voltage removed. This line was only noted in one cell, and it appeared that the alignment agent on the surface was rubbed off on the edges of the indium-tin oxide lines.

3.3 CONCLUSIONS

The overall objective of this task was to define parameters and to develop a seal that was chemically compatible with the reticle manufacturing process. To achieve this objective, three subtasks were defined and investigated: the peripheral LC cell sealant, LC cell fill methods, and the LC fill hole sealant.

The first requirement for this task was to synthesize additional Hughes proprietary LC-R9 materials. Each component was synthesized and recrystallized to obtain 99.8 percent purity, determined with an analytical liquid chromatograph.

Of the three LC peripheral sealing schemes investigated, the dry film sealant with Ablefilm 539 (type II) proved the best approach. The sealant gave excellent results and satisfied the task objective. At no time during tests did the Ablefilm sealant show signs of weakening or reacting with the Hughes proprietary LC-R9 material. Ablefilm 539 (type II) was inexpensive, simple to use and directly compatible with the present LCR manufacturing process.

The method of filling the LCR cell and plugging the fill hole(s) were considered important subtasks to prevent leaks. Of the techniques studied to fill an LC cell, only the one-hole edge fill method was compatible with the reticle manufacturing process. Means of plugging the fill hole were also investigated. Of the schemes studied, epoxy sealants, Ablestik 224-1 or Epoxy Technology 408-3, performed satisfactorily.

Life tests were performed with the Hughes proprietary LC-R9 material using cells constructed of Ablefilm 539 (Type II) with a one-hole edge fill and epoxy fill hole sealant. Tests at +80° and -17°C have surpassed 7000 hours without cell deterioration. Cells with bulk electrodes and continuous AC voltage have surpassed 6200 hours at +25°C without alignment change.

SECTION IV

ELECTRONICS SEAL DEVELOPMENT TASKS

4.1 AIMS AND OBJECTIVES

The main objective of this task was to develop an environmental seal for the electronics required to address and drive individual azimuth and elevation reticle lines. Considerations were given to the selection of a seal compatible with the reticle manufacturing process that would not create opens or shorts of individual reticle lines.

4.2 RESULTS

4.2.1 Considerations and Approach to Electronics Protection

Currently the CMOS electronic driver chips are designed to mount on each LCR glass substrate. To maintain the desired reticle reliability, the driver chips controlling reticle operation must be protected from damage caused by either mishandling or environmental exposure. Mishandling can result in mechanical damage, such as the accidental destruction of wire bonds or devices, and is best avoided by encasing the electronics in a protective package. Environmental damage occurs primarily when moisture in the system allows corrosion to occur, this problem can be controlled by minimizing the opportunities for moisture to be introduced. Thus, it is particularly important to have a knowledge of the moisture-induced failure mechanisms for the CMOS technology used in the electronic driver chips.¹

The primary moisture failure mechanisms of concern to the CMOS devices used on the LCR were

1. Electrical parameter changes caused by motion of charge in or on oxides
2. Aluminum metal corrosion (wire bonds, chip inter-connections).

Increased gate input current, higher gate threshold voltage and increased leakage current are the more important electrical parameters readily changed

¹L. J. Gallace, H. L. Pujol, and G. L. Schnable, "Reliability of CMOS Integrated Circuits," Computer, 11, No. 10, October 1978, pp 6-13.

by charge motion. Both chlorine and the more corrosive sodium ion migration cause electrical parameter changes. Water vapor encountered in a moderately harsh military environment readily contains these ions. Chlorine ions attack aluminum used in integrated circuits for interconnect metallization and wire bonds. For instance, in a matter of a few days after exposure to moisture containing chlorine, the aluminum contacts cannot pass electrical signals. To protect the chip aluminum metallization, inorganic passivation materials are very effective in reducing the possibility of aluminum corrosion. Gold wire is used to replace aluminum wire for bonding, but at increased cost and lower wire rigidity.

Three approaches were investigated for use on the LCR: epoxy encapsulation, protective coatings, and conventional hybrid solder sealing. The selected approach was conventional hybrid solder sealing with new packaging schemes to protect the electronic driver chips. Hybrid solder sealing approach used a mature reliable technology to ensure good electronics seals. An electronics module with all azimuth or elevation driver chips was used to devise two packaging concepts: (1) rigid-flex cabling to physically separate the LC cell from the electronics module or (2) bonding the electronics module to the LC cell.

Epoxy encapsulation was ruled out because of poor repairability, non-compatible curing temperature, unsuitable outgassing materials, and lack of solid data on moisture protection.

Initially protective coatings were investigated on a Hughes-funded IR&D program. Coatings were investigated because they promised to shield the electronic driver chips from particle or moisture contamination (mechanical protection necessary to prevent mishandling). On this contract, protective coatings were investigated further and were found to be incompatible with the present LCR manufacturing process because of poor repairability, a need for multiple coatings, and an aging process that created microcracks in the coatings. The microcracks were judged to leave the electronic chip particularly susceptible to moisture contamination.

The three approaches are discussed next. Electronics protection, repairability, and applicability to the present LCR manufacturing processes are discussed for each approach.

4.2.2 Epoxy Encapsulation

The use of epoxy was considered to obtain a hermetic seal primarily because the circuitry did not have to be coated before encapsulation. Other advantages considered were low process cost, good resistance to shock and vibration, and mechanically strong package not requiring a cover.

Four potential problems associated with epoxy encapsulation of the reticle driver chips were studied:

1. Curing
2. Outgassing
3. Repair
4. Moisture Protection.

No adequate solution to these potential problems was found.

Epoxy compounds that cured well and did not outgass below 80°C were difficult to identify. Outgassing materials were particularly important so as not to attack and destroy the CMOS devices. An effort to investigate epoxy encapsulation materials for use on the LCR was beyond the scope of this contract.

The combination of high humidity levels (>85 percent) and elevated temperature (>100°C) is known to accelerate moisture-induced failure mechanisms in epoxy encapsulated devices. Advances were made in recent years to improve moisture resistance epoxies for semiconductor packaging, but years of testing are required.

Repair of epoxy encapsulated electronics was the last item of concern. Selective etching techniques (chemicals) were difficult if not impossible. Sawing or cutting was not particularly attractive on the reticle. Heating an epoxy above the curing point was another method considered, but temperatures were well above 80°C. Thus, all factors considered, the disadvantages of using epoxy encapsulation on the LCR to protect the driver chips outweighed the advantages.

4.2.3 Protective Coatings

Requirements for a protective coating are given below:

1. To prevent moisture from contacting either the chip surface or chip-to-substrate interconnection wires
2. To not influence chip electrical performance
3. To be simply re-applied after chip or wire repair
4. To be compatible with the present LCR manufacturing process.

Two materials and three coating conditions were investigated on the Hughes IR&D program. The two materials were silicon nitride and parylene "C". The three coating conditions were silicon nitride, parylene "C", and silicon nitride over parylene "C". These coatings had moisture vapor transmission rates that are lower than other materials used in electronic packaging.^{2,3}

The silicon nitride (Si_3N_4) process was a low-temperature photochemical deposition developed by the Hughes Technology Support Division. The temperature of the sample during deposition was never higher than 100°C (65°C minimum), and the technique used ultraviolet light to excite the mixture of silanehydrazine. A mercury vapor resonance-arc source was used to sensitize the molecules with 2537\AA radiation. To achieve sufficient radiation of the gas phase, the system was saturated with mercury vapor. The excited mercury atom collides with the chemicals in the system to generate free radicals. These initiate the chain reactions that lead to the final Si_3N_4 product. A uniform layer of 800 to 1500\AA thick was deposited on exposed surfaces. Repairs of bonds were performed without removing the coating.

²"Low Temperature Deposition of Silicon Nitride Passivation Layers," Hughes Contract No. F33615-76-C-5081, R&D with AFML, 15 February 1977.

³S. M. Lee, J. J. Licaci, "Reliability of Parylene Films," NASA/Electronic Research Center, Anaheim, CA, NDA

Parylene "C" was produced by vapor-phase deposition and polymerization of para-xylylene or its substituted derivatives. Molecular weight was approximately 50,000. Unlike dip or spray coating, condensation coating did not run off or sag. It was not "line of sight" as in vapor metalizing; the vapor coated evenly over edge, points and internal areas. With parylene, the object to be covered remained at or near room temperature and eliminated all risks of thermal damage.

Parylene "C" over silicon nitride was deposited in the same manner as described for each material separately. No special cleaning or handling procedures were needed. Adhesion of the parylene to silicon nitride was generally better than adhesion of the parylenes over bare alumina. To initially evaluate the coatings, 1 x 1 inch HAC PAC test packages were prepared. These contained moisture-sensitive chips and transistors mounted on etched thin film patterns of gold metallized alumina. These moisture chips were made by Panametrics and allowed in MIL-STD-883B, Method 1018. With these chips, the amount of moisture trapped inside a sealed package can be known without having to destroy the package.⁴ 2N2222 transistor chips,* were attached by the silver-filled conductive epoxy, Abelbond 606-2. The moisture chips were attached with non-corrosive epoxy Abelfilm 550. The substrates were bonded in the HAC PACs with Scotchcast 281.

Two sets of test packages were coated with silicon nitride, parylene "C" and combination of silicon nitride over parylene "C". Control packages were also prepared with the coated samples (hermetic packages, plus uncoated and consealed packages). The first set of samples (except hermetic control) were protected mechanically with a silicon rubber gasket and clamped metal lid. The second set of samples (except hermetic control) used on epoxy bonded metal lid.

⁴ M. G. Kovac, et al, "A New Moisture Sensor for in-situ Monitoring of Sealed Packages," Inter. Reliability Physics Symposium, 13 April 1977, Las Vegas, NV.

* Hughes proprietary CMOS chips were not available during IR&D program.

By using the SEM (Scanning Electron Microscope) and EDAX (Energy Dispersive X-Ray Analyzer) when necessary, composition and uniformity of each coating were determined, both before and after environmental tests. Some silicon nitride coated samples showed chlorine ions trapped in the coating, caused by the solvent used to clean the deposition chamber. When the cleaning solvent was changed, the deposited layer closely followed the contour of the sample and was free of nodules. EDAX analysis showed complete absence of contaminants. The parylene "C" coated samples were uniform, but adhesion to the alumina, silicon and gold was marginal, i.e., the coating could be peeled off once a free edge was grasped with a pair of tweezers. The coated samples of parylene "C" over silicon nitride were uniform and free of nodules or bubbles.

The samples were tested according to MIL-STD-810C, Method 507-1, which is the minimum DOD requirement for land-based electronic instrumentation. The test consisted of cycling the samples in 95 percent relative humidity for five cycles (one cycle lasts 24 hours) through the temperature excursion 25 to 60°C. The first set of samples (11 packages) was run for five cycles, as required by the MIL-STD, and tested. The second set of samples (eight packages) was run for 15 cycles with electrical testing every five cycles. All electrical and humidity readings were obtained within 2 hours of removal from the moisture chamber.

The initial test results performed on the Hughes IR&D program are summarized below. Changes in moisture at the silicon chip's surface are given in PPM of water.

Compared to the hermetic package (300 ppm), mechanically protected samples coated with the parylene/ Si_3N_4 combination provided the best protection against moisture penetration (2000 ppm). MIL-STD-883, Method 5008 defines an acceptable moisture level in hybrid packages of 6000 ppm at one atmosphere pressure. Thus the package ambient cannot reach a dew point at a temperature above 0°C.

The silicon nitride coating (2000-5000 ppm) was nearly as effective as the combination coating, whereas only the parylene alone was unsatisfactory (10K to 20K ppm moisture). As expected, the uncoated sample had a very high moisture content (>100K ppm).

The second set of packages was subjected to three times the required number of cycles specified in MIL-STD-810C. A slight improvement in protection was provided by the method of sealing the lid to the package. In this experiment, the clamp and lid assembly was replaced with a lid attached to the base by the Ablefilm 550 epoxy.

The electrical data taken showed that epoxy sealing of the second set of samples resulted in less moisture buildup during environmental exposure than occurred in the lid clamp samples of the first set. The damage to the uncoated samples increased after 3 weeks of exposure, as expected.

For this contract, a plan was formulated to rerun the coating tests using CMOS devices on glass substrates, which were bonded to 14-pin Tekform dip packages. The package scheme was compatible with automatic test equipment. The reasons for this plan were that the previously used packages did not simulate glass reticle plates and CMOS devices were judged to be much more sensitive to moisture than bipolar devices. In addition, the silicon nitride coating was to be applied at temperatures below 100°C, i.e., 75°C.

After 32 sample packages were prepared, several events occurred that completely stopped the tests. First, previously unknown evidence was introduced about an undesirable long-term aging effect in parylene "C" coatings. Coatings of parylene "C" held at room temperature for more than 3 years show that cracking of the material had occurred. Second, experiments indicated process problems associated with silicon nitride deposition at +75°C. Problems also occurred concerning the repeatability of coatings and were considered analogous to sudden loss of yield on a semiconductor wafer line. Finally, experiments to remove the coating, repair a chip, and recoat were not successful.

The coating process was not economical to apply, and the question of process yield was not answered. Thus, the economics plus unsatisfactory test results led to dropping the use of a coating to protect the electronic driver chips on the LCR.

4.2.4 Conventional Hybrid Solder Sealing

Conventional solder sealing is of interest because the technique is widely used in military hybrids to provide mechanical and hermetic protection in a single operation. The procedure consists of metallizing an electronic chip layout on a ceramic substrate, complete with a border for sealing. A Kovar ring this is soldered in place on the substrate border. Next, die attachment and electronic functional tests are completed. Before sealing, the whole assembly is cleaned and a lid soldered on the ring to complete the package. For severe environments, lids with a small hole are used for sealing. The purpose is to pull out the air and refill with a dry inert gas before plugging the small hole. At this point, the whole assembly is gas leak-checked to ensure a long-term hermetic seal.

Conventional hybrid solder sealing techniques cannot be used directly on the LCR glass substrates because of the possibility of ruining the LC cells and cracking the glass substrate from the high temperatures. To use conventional hybrid solder sealing, a new packaging scheme was required to:

1. Overcome the disadvantages of the other electronic sealing methods studied and utilize reliable conventional hybrid sealing techniques.
2. Produce LCR assemblies in the quantities required and at reasonable cost to the government.

Two concepts were studied and described below. Both offer a solid approach to low-cost reliable reticle assemblies with the unique ability to independently fabricate and test high-line yield LC cells and sealed electronics modules.

The first concept uses rigid-flex cabling (RFC) to physically separate the LC cell and electronics module(s) to maximize producibility and maintainability. RFC is a proven and reliable technology for military components suppliers. Several major connector and cable manufacturers are now supplying RFC to the military.

Using the RFC concept, LCR packaging can be modified to fit the needs of each requirement without the need to redesign either the reticle plates or electronics module. Production costs are certainly lower for a large lot of one flexible design rather than for smaller lots of several rigid designs.

A second concept being studied does not physically separate the LC cell from the sealed electronics module. In this case, the sealed electronics module using either hybrid or LSI technology is bonded and wired to a high line yield working LC cell. This approach has the same potential advantages of lower costs, higher overall yield, and improved reliability as the above device that uses RFC. This design is somewhat more rigid in its mechanical structure but has the advantage of not requiring cables.

Both concepts make use of proven, reliable electronic hybrid or LSI technology to ensure good seals. They appear to be applicable to most of the currently deployed fire control systems.

An LCR electronics module designed with standard catalog parts and conventional hybrid sealing technology is shown in Figure 8. The RFC concept incorporated into a LCR package is shown in Figure 9; a bonded electronics module incorporated into a LCR package is shown in Figure 10.

4.3 CONCLUSIONS

The main objective of this task was to develop an environmental seal for the electronics to address and drive individual azimuth and elevation lines. To achieve this objective, a sealing scheme was required that was compatible with the reticle manufacturing process while not creating opens or shorts of individual reticle lines.

Three electronics sealing schemes were rigorously investigated, and the conventional hybrid solder sealing was selected as the best approach. Conventional hybrid solder sealing uses proven and reliable technology to ensure good seals; but to produce LCR assemblies in the quantities required and at reasonable cost to the government, innovative packaging design was also required. The ability to independently fabricate and test a high line yield LC cell and sealed electronics module resulted in low cost reliable reticle assemblies.

Two packaging concepts were devised that use the same electronics module. The first concept used rigid-flex cabling to physically separate the high line yield LC cell and electronics module. Using the RFC concept, LCR packaging can be modified to fit the needs of each requirement without redesigning the reticle plates. The second concept did not physically

separate the LC cell from the sealed electronics. In this case, the sealed electronics module is bonded and wired to a high line yield LC cell. This design is somewhat more rigid in its mechanical structure but has the advantage of not requiring cables.

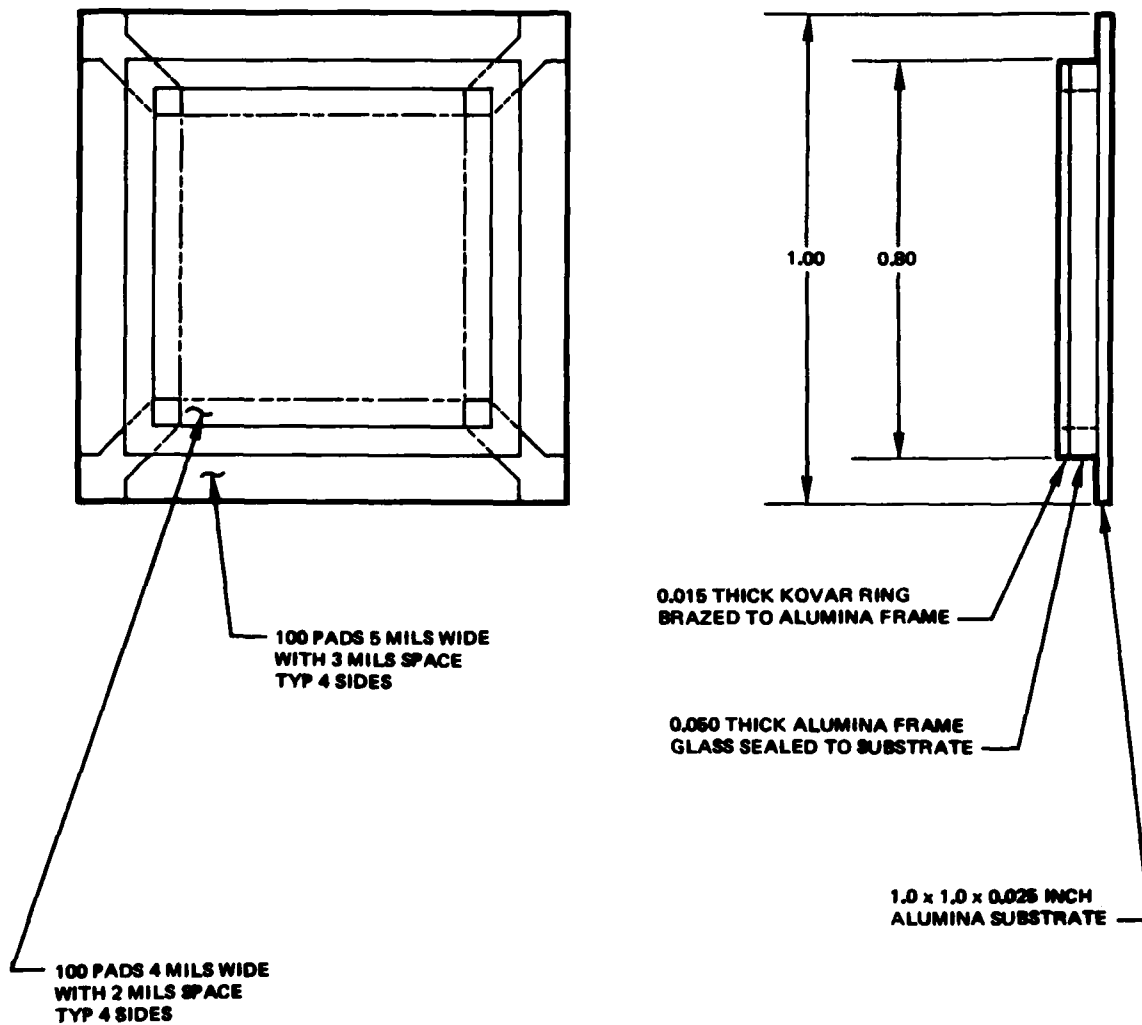


Figure 8. Electronic module

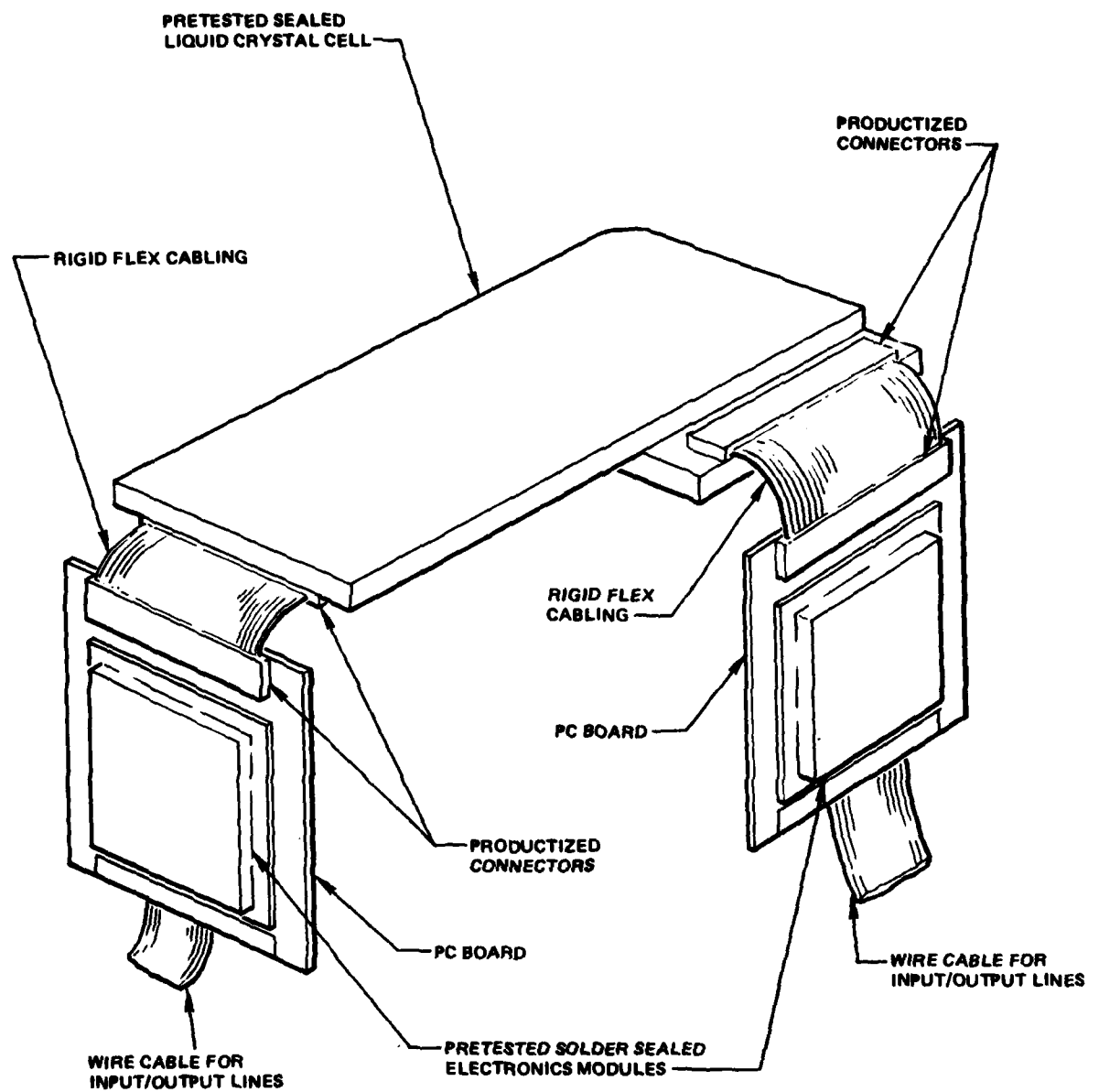


Figure 9. Pretested electronics modules connected to pretested liquid crystal cell via rigid flex cabling.

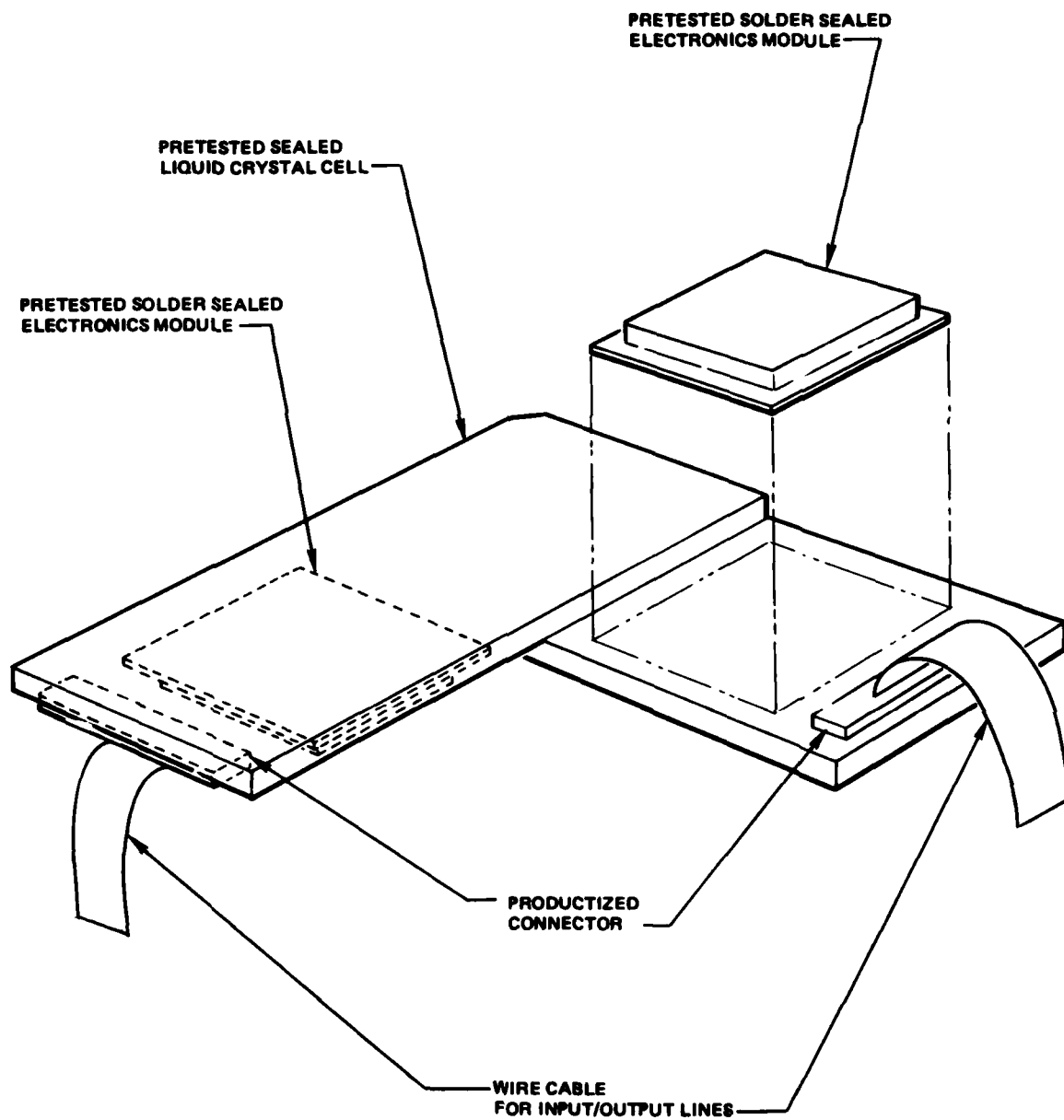


Figure 10. Pretested electronics modules bonded to pretested liquid crystal cell

SECTION V

CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

The techniques developed on the first phase of this two-phase program demonstrated:

1. Techniques to produce 98 percent working electrode lines without repair (100 percent with repair) on LCR plates with a density of 580 lines per inch.
2. That LC cells fabricated with Ablefilm 539 sealant have surpassed 7000 hours at +80 percent and -17°C without cell deterioration. Additional cells with bulk electrodes and continuous AC voltage have surpassed 6200 hours at +25°C without alignment change. Other cells fabricated with Ablefilm 539 showed no signs of weakening when exposed to liquid nitrogen or boiling water conditions.
3. Two packaging concepts for sealing the electronic driver chips, using conventional hybrid sealing technology to maximize producability and maintainability with potentially lower production costs.

5.2 RECOMMENDATIONS

It is recommended that Phase II of this program be funded to incorporate the results of the development tasks into a fieldworthy prototype. Specifically, the goals of Phase II are to:

1. Fabricate a two-axis LCR, incorporating all previous technology with a goal of 100 percent working electrode lines
2. Integrate the LCR into the AN/VSG-2 government furnished tank sight.

It is recommended that additional shock, vibration, and temperature tests be funded for the LCR to prove reliability. In addition it is recommended that an advanced development model integrated into a thermal nightsight, installed in an armored vehicle, be procured to demonstrate improved operational capability. Finally, it is believed that the successful completion of this and concurrent efforts brings the concept of the LCR to the point where full-scale engineering development should be pursued.

APPENDIX A
PROCEDURE FOR OBTAINING HIGH PURITY
LIQUID CRYSTAL COMPONENTS

The procedure established for obtaining high purity LC components began with the synthesis of each sample. After synthesis and recrystallization, a sample of each compound was run through an analytical liquid chromatograph (Waters ALC/GPC Model 502/401). This instrument used an activated silica column to separate components in a chemical sample according to their polarity. Each constituent of the injected sample ($\sim 10^{-5}$ gram) was analyzed by absorbance of 254 nm radiation. As each resolved constituent of the sample leaves the column, its presence was detected by absorbance as a function of time. Each component then appeared as a peak, whose area was proportional to the amount of that component. The relative positions of these peaks was governed by the retention time (polarity) of each unknown in the column (time to leave the column). Using this technique, impurity levels of 0.0028 can be detected in a 10^{-6} gram sample.

Although most of the components of mixture LC-R9 were purified by recrystallization to levels of 99.8 percent purity, a few components reached levels of only 97 percent. These materials contained impurities that had chemical structures similar to each desired compound. This structural similarity made recrystallization difficult; therefore, another method was required. A preparative liquid chromatograph (Waters Model 500) was used to separate the desired material from the impurities. This liquid chromatograph was similar to the analytical-type except quantitative accuracy was sacrificed for loading capability.

First, the retention times were determined for the desired sample and impurities (by peak separation and height) from the analytical chromatograph. With the retention times known, a large sample (~ 10 grams) was injected into the preparative chromatograph. By waiting the appropriate times, each component of the sample was anticipated and separated, as each peak (component) began to appear (leave the column). In this way, the impurities were "cut away" from the desired material. Following this purification step, quantitative purity was determined on the analytical chromatograph. Using

this technique, purity levels of 99.8 percent were obtained for all the components of LC-R9.

APPENDIX B
ABLEFILM 539 DATA



ABLESTIK LABORATORIES

833 WEST 182ND STREET, GARDENA, CALIFORNIA 90248 • (213) 321-6252

ABLEFILM
539

CONTROLLED FLOW FILM ADHESIVE FOR BONDING LC PLATES

DESCRIPTION

This data sheet describes a series of specially formulated epoxy adhesive preforms for use in liquid crystal applications.

These adhesives have been used successfully for bonding LC plates together and have not caused any contamination or "poisoning" of the LC fluids.

Supplied as die-cut preforms to fit an exact LC configuration, they can be made in one-half mil increments ranging from 0.0005 to 0.003 inches thick. While Mylar is generally used as the adhesive carrier-spacer, other material can be used depending upon a specific customer requirement.

Because liquid crystals generally are made with either nematic or cholesterol fluids and additives that tend to differ between manufacturers, ABLEFILM 539 has been created to provide a versatile adhesive preform through the use of different basic resins and curing agents.

In the event that the types of ABLEFILM 539 described are not suitable for use with a potential customer's particular LC formulation, ABLESTIK will tailor a type to insure product compatibility.

ADVANTAGES

- | | |
|----------------------------|---|
| Tack Free Film: | ABLEFILM 539 film adhesive eliminates the messy handling, clean up and dermatitis characteristic of conventional paste or liquid epoxy adhesives. |
| Low Condensible Volatiles: | ABLEFILM 539 produces virtually no condensible volatiles to contaminate or corrode circuitry. |
| Controlled Flow: | ABLEFILM 539 has been especially formulated to prevent excessive flow during the cure process thereby minimizing adhesive penetration into the LC display area. However, adhesive flow slightly increases as the type number increases. |

Manufactured by ABLESTIK ADHESIVE DIVISION, 833 West 182 Street, Gardena, Calif. • Telephone (213) 321-6252

TYPICAL PROPERTIES

ABLEFILM 539 - TYPE 1*

Adhesive Type	Modified Epoxy	Cure:	1 Hour @ 175°C ½ Hour @ 200°C
Lap Shear Strength (alum to alum)	5,000 psi	Storage:	6 months at room temperature (25°C) 1 year at 0°C or colder

ABLEFILM 539 - TYPE 11*

Adhesive Type	Epoxy	Cure:	2 Hours @ 125°C 1 Hour @ 150°C ½ Hour @ 175°C
Lap Shear Strength (alum to alum)	3,000 psi	Storage:	6 months at room temperature (25°C) 1 year at 0°C or colder

ABLEFILM 539 - TYPE 111*

Adhesive Type	Epoxy	Cure:	1½ Hours @ 160°C 1 Hour @ 175°C 1 Hour @ 200°C
Lap Shear Strength (alum to alum)	3,000 psi	Storage:	1 year at -40°C 30 days at room temp. (25°C)

*NOTE: This material does not contain amines, acids or anhydrides.

APPLICATION

1. Place film between surfaces of parts to be bonded.
2. Assemble parts and provide continuous pressure sufficient to maintain intimate contact between adhesive and bonding surfaces. Dead weight or spring clamps have been found suitable.
3. Place assembled package in oven and heat at desired cure temperature for recommended period of time.

CAUTION: This product may cause skin irritation to sensitive personnel. If contact with skin occurs, wash affected area immediately with soap and water.

DISCLAIMER: All statements, technical information and recommendations contained herein are based on tests we believe to be accurate, but the accuracy or completeness thereof is not guaranteed, and the following is made in lieu of warranty express or implied; Seller and manufacturer's only obligation shall be to replace such quantity of the product proved to be defective. Neither seller nor manufacturer shall be liable for any injury, loss or damage, direct or consequential, arising from the use or inability to use the product. Before using, user shall determine the suitability of the product for his intended use, and user assumes all risk and liability whatsoever in connection therewith. No statement or recommendation not contained herein shall have any force or effect unless in an agreement signed by officers of seller and manufacturer.

APPENDIX C

PROCEDURES FOR ASSEMBLING LIQUID CRYSTAL CELLS

Nylon gloves were used for all operations. Special care was used to ensure that no solvents came into contact with the gloves during substrate handling. Reticle substrates were placed in a glass rack and rinsed with acetone and chloroform. Then they were ultrasonically cleaned in acetone and also in chloroform for about 5 minutes each. The substrates were rinsed with chloroform and finally vapor dried in isopropanol using the vapor degreaser.

Different surface preparations were required to control molecular orientation on the electrode surfaces for AC and DC driven cells. The procedure for preparing DC driven LCR cells was to rub the electrodes without special coatings. The cleaned electrode surface was rubbed with a pad using light to medium pressure. The pad was prepared by wrapping Kim wipes uniformly around a ruler or microscope slide, and the surface was kept clean. A uniform continuous motion was used over the active reticle display area; parallel strokes were used for each substrate. The rubbing direction was parallel to the reticle lines (i.e., the rubbing directions on the azimuth plate and elevation plate were perpendicular to each other).

The procedure for preparing AC driven cells was to treat the electrode surfaces with an alignment agent. A polyvinyl alcohol, SA-72 (American Liquid Crystal Chemical Co.), was used for surface treatment. The application is given below:

Cleaned substrates were rinsed with DI water while spinning at 2000 rpm for 60 seconds. The active reticle area was then coated with a diluted mixture of three parts DI water to one part SA-72 and spun at 2000 rpm for 60 seconds. The coating was baked at 110°C for 2 hours in a clean oven. The baked coating was rubbed in the same manner for DC cell preparation.

After surface preparation, Ablefilm 539 Type II (0.5 mil mylar and 0.1 epoxy in B-stage coated on both sides of the mylar) was cut to the desired shape. The mylar sheet acted as an adhesive carrier and also as a controlled thickness spacer. The preform was cut with a break water type channel for filling the liquid crystal, as shown in Figure 7. The preform was positioned on one substrate without touching the active area. A counter electrode substrate was placed in position over the first substrate, and alignment markings were registered to ensure correct placement. This operation was performed best under a coaxial microscope. The substrates were clamped together on all sides and the assembly placed in a clean oven at 150°C with nitrogen flow for at least 1.5 hours. Later in the assembly it was removed from the oven and left to cool before removing the clamps.

Next, a small dish was filled with LC-R9 liquid crystal material (about 1 gram) and placed in the filling chamber. The assembled LCR cell was held with a paper clamp that was attached to a metal rod which in turn run through a rubber stopper in the top of the filling chamber. The cell was positioned such that the fill hole was just above the surface of the liquid crystal material. After the chamber was evacuated to at least 50 μ , the holding rod was lowered to immerse the LCR fill hole into the LC material and start filling the cell. When the cell was about one third full, the vacuum was slowly released to complete filling the cell.

NOTE: Fresh LC material was outgassed by evacuating the filling chamber for at least 3 hours.

Finally, the filled cell was removed from the filling chamber and liquid crystal material cleaned off from the fill hole area using Kimwipes or tissues. A solvent was not used because of possible contamination in the LC material. The fill hole was plugged with either 224-1 or 408-3 supplied by Ablestik and left to cure at room temperature.

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